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# Fabrication of form stable NaCl-Al<sub>2</sub>O<sub>3</sub> composite for thermal energy storage by cold sintering process

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**Abstract** A form stable NaCl-Al<sub>2</sub>O<sub>3</sub> (50-50 wt-%) composite material for high temperature thermal energy storage was fabricated by cold sintering process, a process recently applied to the densification of ceramics at low temperature 300°C under uniaxial pressure in the presence of small amount of transient liquid. The fabricated composite achieved as high as 98.65% of the theoretical density. The NaCl-Al<sub>2</sub>O<sub>3</sub> composite also retained the chloride salt without leakage after 30 heating-cooling cycles between 750°C–850°C together with a holding period of 24 h at 850°C. X-ray diffraction measurements indicated congruent solubility of the alumina in chloride salt, excellent compatibility of NaCl with Al<sub>2</sub>O<sub>3</sub>, and chemical stability at high temperature. Structural analysis by scanning electron microscope also showed limited grain growth, high density, uniform NaCl distribution and clear faceted composite structure without inter-diffusion. The latent heat storage density of 252.5 J/g was obtained from simultaneous thermal analysis. Fracture strength test showed high sintered strength around 5 GPa after 50 min. The composite was found to have fair mass losses due to volatilization. Overall, cold sintering process has the potential to be an efficient, safe and cost-effective strategy for the fabrication of high temperature thermal energy storage materials.

**Keywords** cold sintering process, composite fabrication, thermal energy storage, phase change materials

## 1 Introduction

The growing demand for renewable energy supply, waste heat recovery and efficient utilization of domestic energy suggests the need for latent heat thermal energy storage (TES) systems using phase change materials (PCMs) to be

developed that can absorb and release large amount of latent heat [1–3]. Therefore, development of reliable, sustainable and affordable thermal energy storage system using PCMs considering growing energy demand, amount of industrial energy losses and abundance renewable sources is the most desirable. PCMs store latent heat as they change phase usually from solid state to liquid state at a temperature that matches the energy supply sources [4,5]. The energy storage capacity potential of these materials at constant temperature is about 5–14 times higher than sensible heat storage materials as reported in numerous literatures [6]. PCMs are classified as organic or inorganic materials applicable in different temperature regions. Organic PCMs are characterized by absence of phase segregation, thermal stability at low temperature, non-corrosive, readily available and nontoxic. This is in addition to a low and unique phase change temperature within the human comfort living temperature zone [3,7]. However, organic PCMs are limited as they decompose at higher temperature and generally characterized by low thermal conductivity [7]. Beyond the decomposition temperature of organic PCMs, inorganic PCMs such as salts, salts eutectics, metals, alloys and salt hydrates are suitable TES materials [7,8]. Pure inorganic salts and their eutectics, in particular the eutectics of chloride salts, have been widely investigated as they are cheap, readily available with high thermal storage capacity applicable in the high temperature region [2,8,9]. Jiang et al. [10] studied the eutectic Na<sub>2</sub>CO<sub>3</sub>-NaCl salt and concluded that it is a promising material when used in CO<sub>2</sub> environment or properly encapsulated at temperature below 700°C. It was also found that a ternary chloride eutectic mixture of NaCl-CaCl<sub>2</sub>-MgCl<sub>2</sub> in a temperature range of 550°C to less than 650°C was thermally stable by investigations using TGA and DSC techniques respectively [11]. For the encapsulation of pure inorganic salts, the conventional high temperature sintering method can address thermal stability challenges and also achieve excellent resistance to corrosion [2,12]. However, the energy consumption during

the sintering process is significant and the PCMs are limited due to high sintering temperature [13]. Similarly, the one step chemical synthesis method reported by Zhu et al. [2] also requires drying for 24 h and heating at temperature of 500°C.

To address the high energy consumption and limitation of PCM selection, an alternative method for the encapsulation of pure inorganic salts and their eutectic is cold sintering process (CSP), an emerging low energy requiring, and inexpensive technology recently applied to the densification of several ceramic materials at low temperature  $\leq 300^\circ\text{C}$  [13–15]. CSP involves dissolution-precipitation aided by transient liquid and sufficient temperature under uniaxial pressure. The approach has not been reported for TES materials fabrication, more so for high temperature application, a region that received less research attention [16]. One of the limitations of CSP is the incongruent, extreme or absolute insolubility of the material to be sintered in the transient liquid. Pure  $\text{Al}_2\text{O}_3$  is insoluble in water and pure NaCl is characterized by extreme water solubility, limiting their densification using CSP. However recent understanding of NaCl as sintering aid of  $\text{Al}_2\text{O}_3$  at a low temperature for dielectric application [17] has open the possibility of extending the approach to other application areas. In the present paper, we demonstrated that CSP was suitable to fabricate NaCl- $\text{Al}_2\text{O}_3$  composite with excellent thermal stability and corrosion resistant for high temperature TES applications.

## 2 Experimental

### 2.1 Raw materials

Raw analytical grade aluminium oxide alpha phase solid powders (purity: 99.95% purity; size: 0.2–0.45 micron) and sodium chloride solid powders (purity: 99.5%; average size: 1.2 microns) were purchased from Alfa Aesar, UK. Deionised water was obtained in the lab.

### 2.2 CSP of NaCl- $\text{Al}_2\text{O}_3$ composite cold sintering process

Initially, 0.6 g sodium chloride solid powders were firstly moisten with 0.15 g deionized water and mixed thoroughly with pestle and mortar for about 1 min until uniformly wetted, and then mixed with 0.6 g of  $\text{Al}_2\text{O}_3$  to form a uniform semi solid paste. The mixture was then transferred into 13 mm heatable die to carry out the CSP fabrication. To identify desired sintering conditions, first group of mixtures were hot pressed at constant temperature of 120°C, with different pressure ranging from 100 to 500 MPa but same holding time of 50 min. Second group of mixtures were hot pressured under constant uniaxial pressure of 400 MPa, with different temperature ranging from 80°C to 140°C but same holding time of

50 min. Third group of mixtures were hot pressed at constant temperature of 140°C and constant pressure of 400 MPa, but with different holding time of 10, 30, 50, 60 and 90 min. All the resulting pellets were dried in an oven with maintained temperature of 100°C for 2 h to ensure residual water if any was removed.

### 2.3 Characterization

The dried composite pellets were characterized for their simple sintering monitors (density, and grain size), phase composition, microstructure, thermal and mechanical properties. The bulk density was determined by dividing the measured pellets mass by its volume obtained from its dimensions. Meanwhile, relative density is the ratio of the measured bulk density of the pellet to the theoretical density of the composite which is 2.9082 g/cm<sup>3</sup> determined from Eq. (1) below:

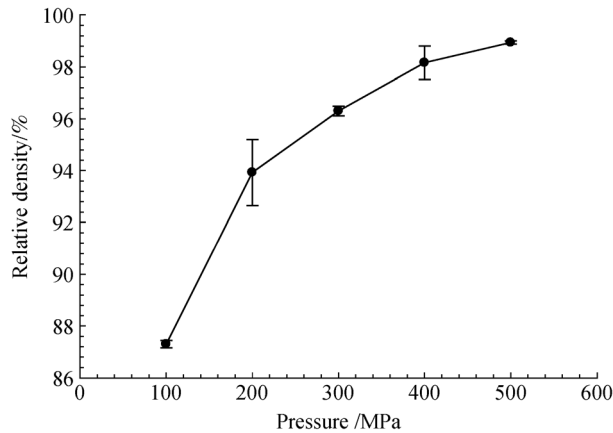
$$\rho_T = \frac{W_1 + W_2}{\frac{W_1}{\rho_1} + \frac{W_2}{\rho_2}}, \quad (1)$$

where  $\rho_T$  is the theoretical density of the composite,  $W_1$ ,  $W_2$ ,  $\rho_1$  and  $\rho_2$  are respectively the weights and densities of NaCl and  $\text{Al}_2\text{O}_3$ .

The microstructural of the sintered samples were observed using scanning electron microscopes (XL30 FEG E-SEM and TM 3030 Plus). The grain size of the composite pellets was further analysed using freely available software image J. The composite phase structure and thermal properties were studied using X-Ray Diffractometer (EVA 4.2.2) and simultaneous thermal analysis (STA) (NETZSCH STA 449 F3 Jupiter®). Strength test of the composite pellets at ambient temperature was performed using universal testing machine (LS100 plus, Lloyd Instruments) according to the Brazilian method reported by Randall [18]. In the thermal cycling test, the composite pellets were arranged in high temperature aluminium oxide square crucible of 12 cm × 12 cm size, which were then placed inside furnace and subjected to 30 heating-cooling cycles between 750°C (1 h dwelling time) and 850°C (1 h dwelling time) at heating and cooling rate of 10 and 1°C/min, respectively.

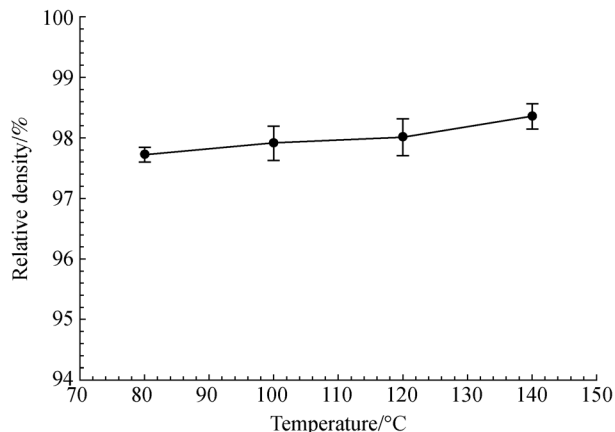
## 3 Results and discussion

Figure 1 shows the density variation with sintering pressure of the NaCl- $\text{Al}_2\text{O}_3$  composite sintered at 120°C with a holding time of 50 min. It revealed that sintered density increases first sharply and then slightly with the increase of pressure, and 87% and 94% of the theoretical relative densities could be achieved under uniaxial pressure of 100 and 200 MPa. The influence of pressure is further demonstrated by the absence of overlap in all the



**Fig. 1** Relative density variation as a function of sintering pressure of cold sintered NaCl-Al<sub>2</sub>O<sub>3</sub> composite at 120°C and with a holding time of 50 min.

error bars of 95% confidence interval. Figure 2 shows the densities variation with sintering temperature at 400 MPa with a same holding time of 50 min. It reveals that relative density of the sintered composite reaches 97.6% at extraordinary low temperature of 80°C. This is even higher than 96% relative density reported by Induja and Sebastine [18] at temperature of 120°C and uniaxial pressure of 200 MPa for dielectric application. Comparably, it also indicates that sintering pressure plays a more important role than sintering temperature on the sintered densities. Though, the influence of sintering temperature and pressure is apparent but unveiling detail fundamental mechanism of NaCl-Al<sub>2</sub>O<sub>3</sub> could provide a more detail fundamental understanding for the fabrication of higher temperature TES materials. Towards the theoretical limit, densification is favoured by time and temperature characterised to be mainly microstructural rather than bulk property changes as observed in our work and also in a reported literature [17].



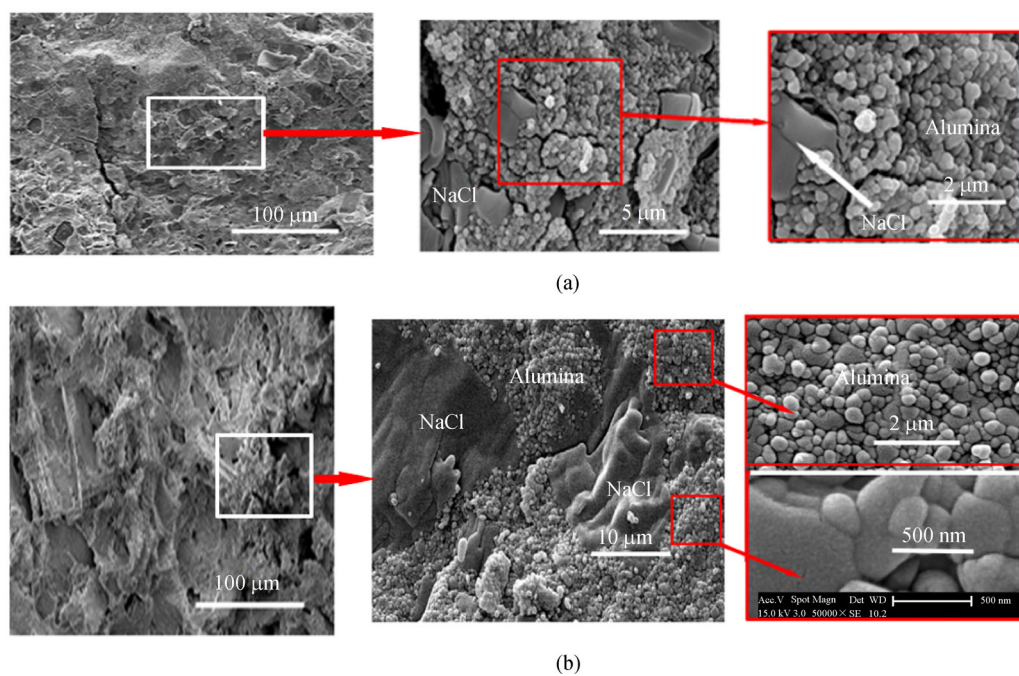
**Fig. 2** Relative density variation as a function of temperature of cold sintered NaCl-Al<sub>2</sub>O<sub>3</sub> composite at 400 MPa with a holding time of 50 min.

The fractured surface SEM images of the composite surface sintered at 120°C, 200 MPa and 140°C, 400 MPa respectively in Figs. 3(a) and (b) show that NaCl was sintered into single monolithic particles to full density overcoming its densification limitation due to extreme solubility in water. The fractured surface image further reveals a clear faceted structure of encapsulated NaCl having clear boundary with Al<sub>2</sub>O<sub>3</sub> interface without inter-diffusion. The observed highly dense structure of the composites is, therefore, consistent with reported high relative density in this study. Although, large grain growth of NaCl is observed but Al<sub>2</sub>O<sub>3</sub> particles are much smaller than the initial powder particles with pores and sharp crack more pronounced along the grain boundary of the composite cold sintered at 120°C and 200 MPa. The decrease in alumina particle size may possibly due to its solubility in NaCl and pressure effect causing chloride ion to break the stable oxide structure into smaller particles. Therefore, it is evident that less surface area and enhanced neck growth promoting microstructural changes are observed in the sintered composite at 140°C in Fig. 3(b).

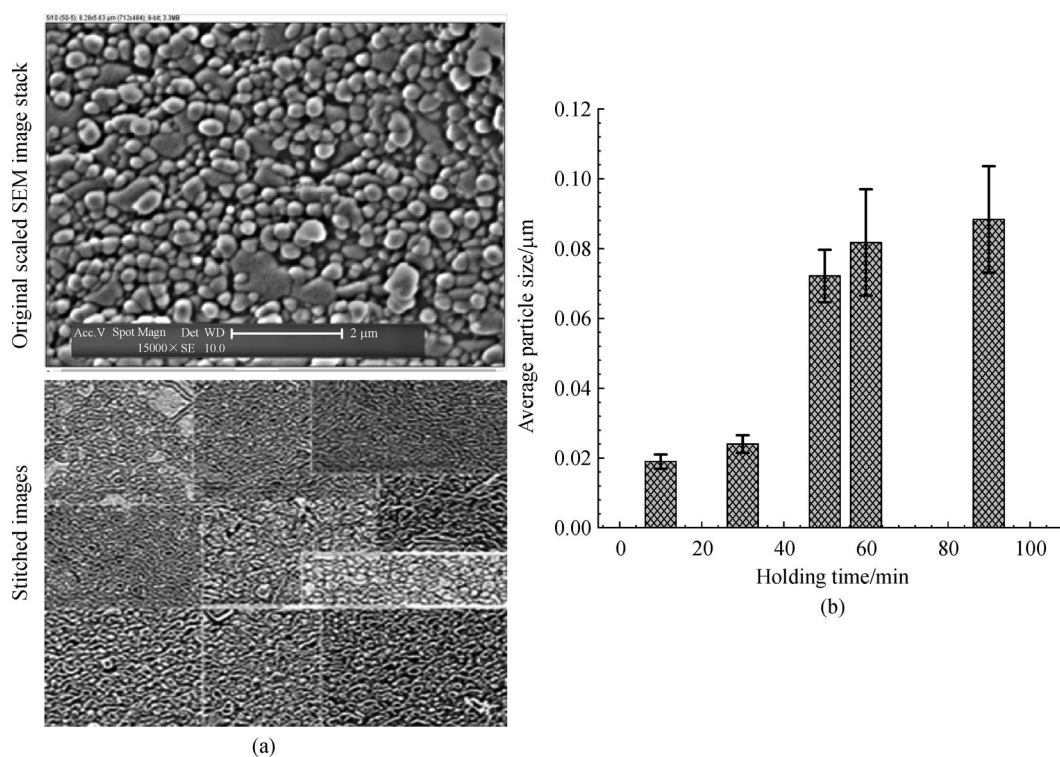
Figure 4 presents detail particle size analysis and the corresponding average size distribution of the sintered NaCl-Al<sub>2</sub>O<sub>3</sub> composite at 140°C and 400 MPa with different holding times. The high relative density achieved and very low average Al<sub>2</sub>O<sub>3</sub> particle size ranging from 0.0189 to 0.089  $\mu$ m different from starting particle size over holding time of 10–90 min revealed the occurrence of cold sintering. This is an indication of the composite fabrication compatibility to the CSP as new grains were formed with limited growth. The observed scenario is linked to the absence of grain coarsening common in the conventional sintering approach [15] or a result of intermediate amorphous phase suppressing boundary diffusion [19]. The observed appearance of new particle sizes in a nano-scale could also be linked prior to a more detail fundamental study to the breaking down of the oxide bond of Al<sub>2</sub>O<sub>3</sub> by high Cl-concentration present in a neutral solution. The Cl- was adsorbed in the Al<sub>2</sub>O<sub>3</sub> crystal as reported in a study of dissolution rate of the Al<sub>2</sub>O<sub>3</sub> passive film in near neutral solution containing NaCl [20].

It is interesting to note that the observed new grains in this study is contrary to the reported alumina final grain size of 2.6  $\mu$ m sintered at 1570°C for 4 h by conventional thermal sintering [21]. Therefore, result obtained in this study shows that using CSP under specified conditions, a polycrystalline material with preserved or controlled crystallite sizes could be achieved as reported in a related study [19].

Figure 5 shows the phase and thermal analysis using X-ray diffraction (XRD) to investigate both chemical compatibility and stability before and after thermal cycling (at PCM melting point) in addition to unveiling the thermal characteristics of the CSP made composite. In terms of phase purity, Fig. 5(c) shows that CSP fabricated sample before thermal cycling contains the phases of Al<sub>2</sub>O<sub>3</sub> and



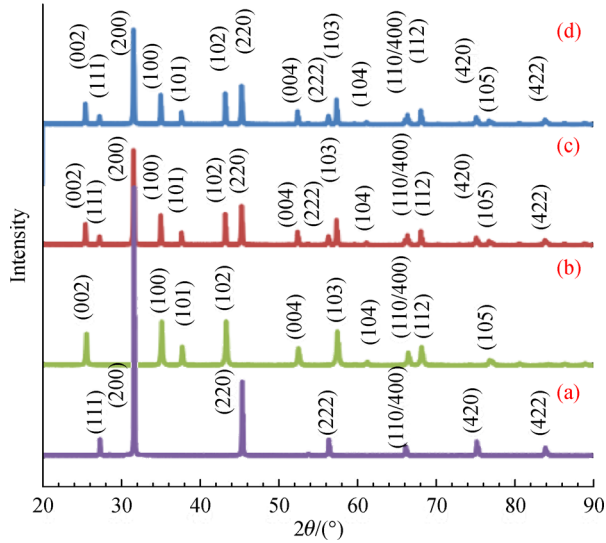
**Fig. 3** Fractured surface SEM images of NaCl-Al<sub>2</sub>O<sub>3</sub> composite cold sintered (a) at 120°C, 200 MPa and 50 min, (b) at 140°C, 400 MPa and 50 min.



**Fig. 4** Cold sintered NaCl-Al<sub>2</sub>O<sub>3</sub> composite average particle size analysis: (a) stitched and original scaled SEM image stack, and (b) average particle size distribution of sintered NaCl-Al<sub>2</sub>O<sub>3</sub> composite.



NaCl only in comparison to the starting powder phases shown in Figs. 5 (a) and (b) where all peaks correspond to the peaks of NaCl and  $\text{Al}_2\text{O}_3$  phases only. NaCl and  $\text{Al}_2\text{O}_3$  XRD peaks were indexed for cubic and hexagonal systems respectively. Therefore, in the absence of any new phase, it is apparent that in the presence of small amount of water (25 wt-% of NaCl),  $\text{Al}_2\text{O}_3$  powder has congruent solubility in NaCl powder. In addition, comparison of Figs. 5(c) and (d) also shows that the phase structure of the composite pellet powder remains the same after 30 cycles. It further suggests that the composite materials fabricated by CSP in this study are chemically compatible and remain stable at

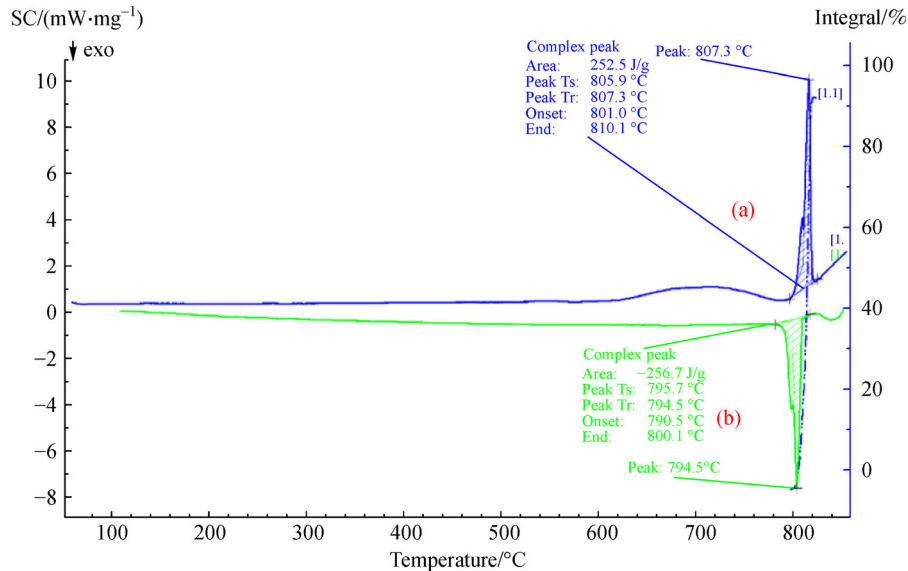


**Fig. 5** XRD Pattern of pure powders and NaCl- $\text{Al}_2\text{O}_3$  composites before and after thermal cycling: (a) NaCl, (b)  $\text{Al}_2\text{O}_3$ , (c) NaCl- $\text{Al}_2\text{O}_3$  composite before thermal cycling, (d) NaCl- $\text{Al}_2\text{O}_3$  composite after thermal cycling.

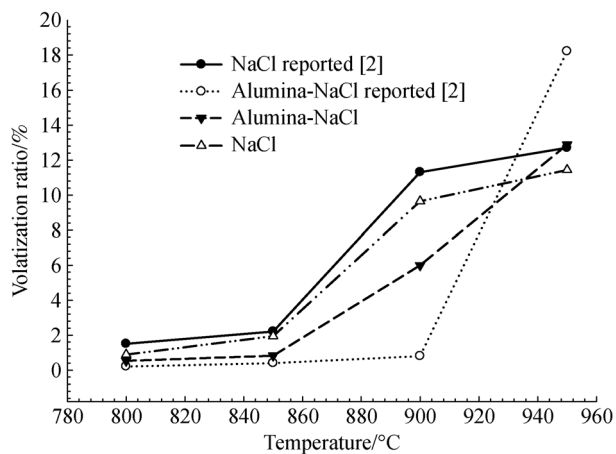
high temperature. Therefore, raising sintering temperature and pressure in this study could only enhance the densification of the ceramic  $\text{Al}_2\text{O}_3$ . These fundamental variables have no effect on its solubility as reported to be the case in the CSP of ceramics sparingly soluble in water [19]. Although, there was no additional phase detected in both the phase and structural analysis at the grain boundaries, future analysis should focus on the role of water and more detail characterization techniques to confirm if additional phase exists in the presence of low or high amount of water.

Figure 6 shows STA curves of the composite (50 wt-% NaCl) with melting and solidification phase change enthalpy of 252.5 and 256.7 J/g respectively. The result also shows that the phase change enthalpy, on set and end temperatures are quite close and only 4.2 J/g variation was observed between the melting and solidification enthalpy curves. This is good and even higher than the composite fabricated from one step chemical synthesis which has a phase change enthalpy of 362 J/g with 75.1 wt-% NaCl [2]. This demonstrates the compatibility of CSP approach for TES material fabrication over wide range of application temperature in terms of composite chemical compatibility, thermal stability and thermal storage capacity. This is competitive and could achieve higher performance than existing methods via material integration at low temperature range.

Figure 7 shows the volatilization result of the pure NaCl and its composite with  $\text{Al}_2\text{O}_3$  over a range of temperature of 800°C–950°C at heating rate of 10°C/min and a dwelling time of 30 min. The results from this study agree well and shows similar trend in comparison to the reported volatilization of the NaCl and NaCl- $\text{Al}_2\text{O}_3$  prepared by one-step chemical synthesis method [2]. In both studies, the mass loss observed in the composite pellet

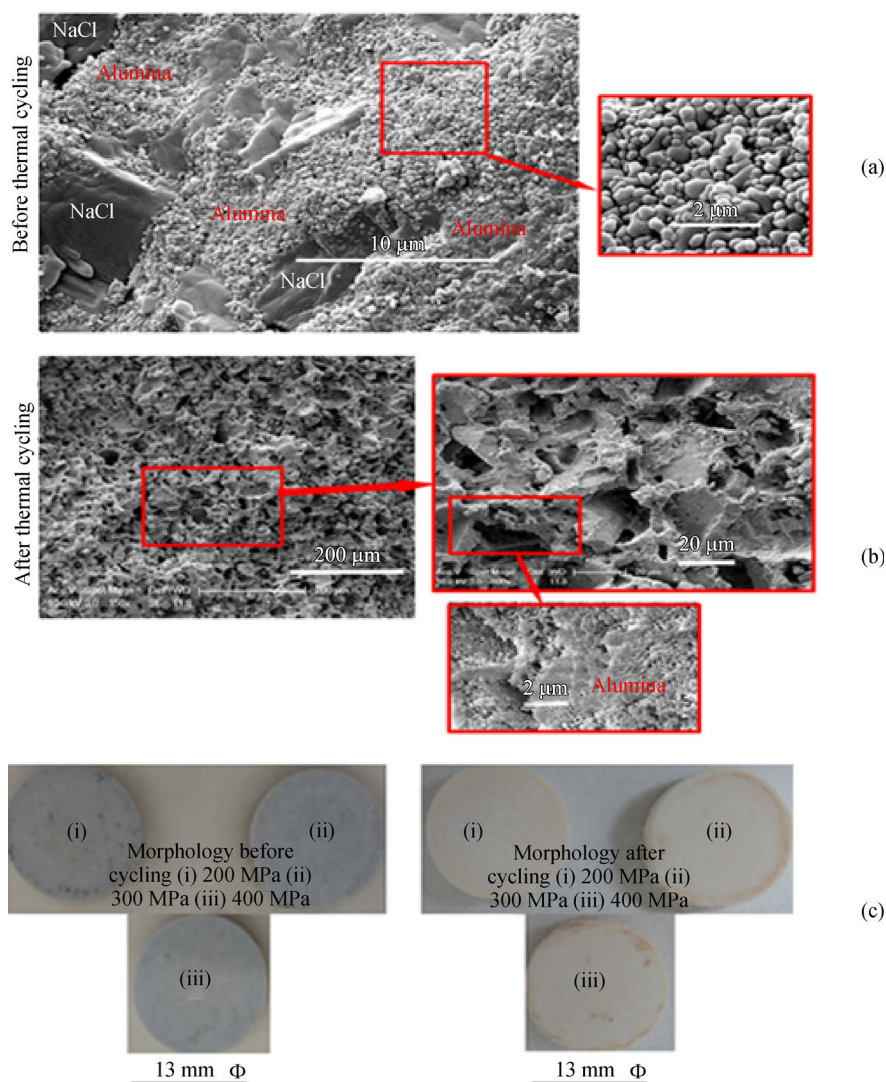


**Fig. 6** STA (a) melting and (b) solidification segment curves of NaCl- $\text{Al}_2\text{O}_3$  composite material fabricated at 140°C, 400 MPa with a holding time of 50 min.



**Fig. 7** Pure NaCl and NaCl- $\text{Al}_2\text{O}_3$  composite volatilization ratio at different temperatures.

was small compared to pure salt in the temperature range of 800°C–900°C. However, there are differences in magnitude of volatilization ratio and the range of temperature within which both composite and pure salt have minimum volatilization. In this study, mass loss of composite is smaller than observed in pure salt up to 850°C beyond which it gradually loses its capacity of minimizing exposed contact surface area with surrounding at high temperature. The increased volatilization at 900°C observed in this study is higher than that reported in [2] could be linked to the dwelling time of 30 min allowed at the chosen set temperatures of this study. Further observation at 950°C was a mass loss of pure salt lower than that of the composite pellet in both studies, very likely due to smaller specific surface area of melted pure salts than composite. For the potential TES application, the volatilization results of NaCl- $\text{Al}_2\text{O}_3$  composite revealed its greatest resistance



**Fig. 8** Microstructure and pellet morphology of NaCl- $\text{Al}_2\text{O}_3$  composite fabricated at 140°C with holding time of 50 min: (a) microstructure before thermal cycling, (b) microstructure after thermal cycling, and (c) pellet morphology before and after thermal cycling.

between 800°C–850°C, indicating a preferable temperature range.

Figure 8 shows details of fractured surface SEM structural images and morphology of the composite pellets before and after thermal cycling with 30 heating-cooling cycles between 750°C–850°C. There was a stable structure and good morphology before cycling. However, after 30 complete thermal cycles, the composite pellets sintered at 140°C under uniaxial pressure of 400 MPa was observed to be stable without leakage, while appeared sharp cracks were observed on pellets sintered at 120°C with sintering pressure of 200 and 300 MPa. Exactly the same observations were found despite holding 24 h at 850°C in one of the thermal cycles.

Generally, physical salt (NaCl) leakage was not observed after repeatable thermal cycling as shown in the pellet images in Fig. 8(c). The observed brightness of the pellet after thermal cycling is because of repeated melting and re-crystallization of the salt at sufficiently high temperature responsible for uniform surface appearance of the composites. However, appearance of a sharp crack and loss of mechanical strength was consistently observed in the sample pellets when the sintering pressure is low. Meanwhile, there was a significant change in the fractured surface structure after thermal cycling, characterized by open pores presumed to be the positions of NaCl which moves across preserved  $\text{Al}_2\text{O}_3$  structural frame or because of the salt volatilization. The latter is also evident in the observed volatilization study of NaCl at various temperatures.

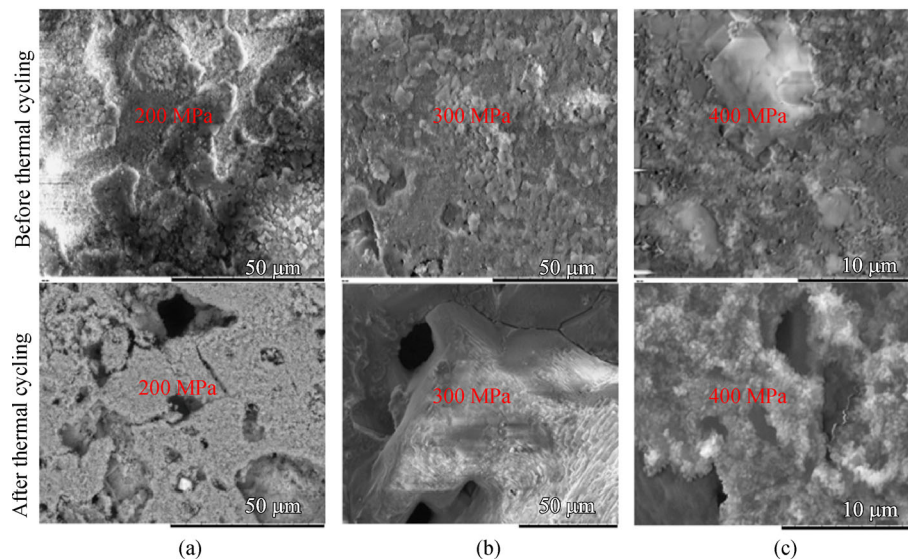
It is important to note that influence of pressure has the greatest contribution to the bulk property (density) increase of the NaCl- $\text{Al}_2\text{O}_3$  composite. Although high sintering density is achievable with low sintering pressure, Fig. 9

shows that after thermal cycling samples sintered at or below 300 MPa lost completely their initial surface structure. On contrast, for samples sintered at 400 MPa, not only structure is maintained but phase and chemical stability obtained from XRD is also reaffirmed by the energy dispersive X-ray spectroscopy (EDS) analysis, as shown in Fig. 10 that only the initial starting material elements (Na, Al, Cl and O) are present in the composite after thermal cycling.

In addition to the phase, structure and chemical compatibility, the composite also has good physical strength as revealed by the strength test. Figures 11(a,b) show that the relative density and sintered strength increases with the increase of holding time in the CSP process. It is apparent that over 4000 MPa of sintered strength is achieved with holding time of 50 min. The change of sintered strength between 10 and 30 min is not very evident but the increase is very clear when holding time increases to 60 and 90 min. Also, as shown in Fig. 11(c) the logarithm of the sintered strength is linearly related to the logarithm of fractional density which agrees with the results reported by Randall et al. [18].

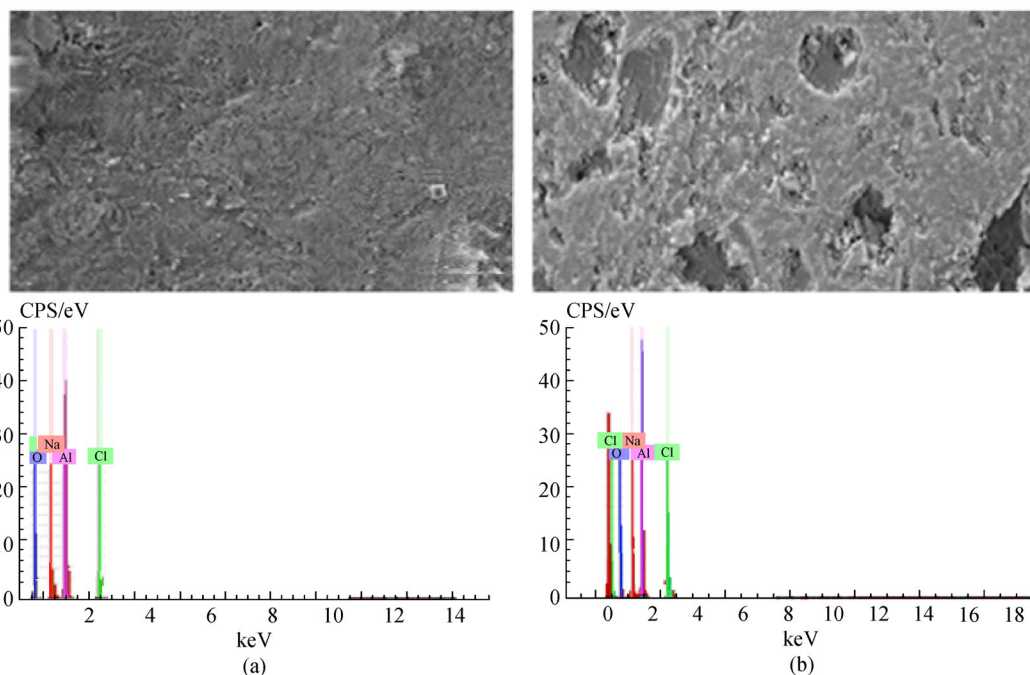
## 4 Conclusions

High temperature NaCl- $\text{Al}_2\text{O}_3$  (50-50 wt-%) composite material for thermal energy storage with large phase change enthalpy of 252 J/g was fabricated at extraordinary low temperature by cold sintering process. The average particle size of  $\text{Al}_2\text{O}_3$  under the observed conditions was in the range 0.0189–0.089  $\mu\text{m}$ , much lower than the starting powder particles size, demonstrating CSP role in the densification of metal oxide supports with chloride salt in

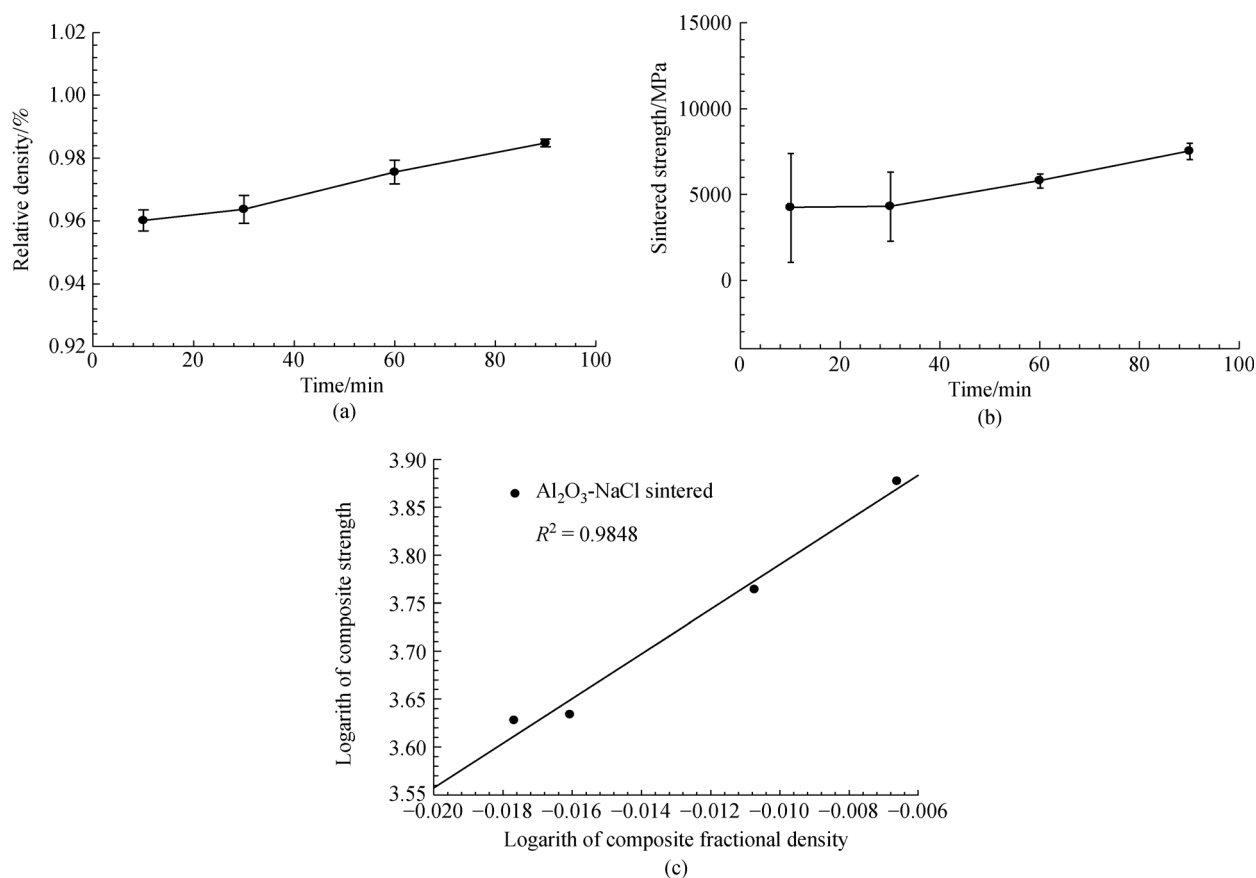


**Fig. 9** Fractured surface SEM images of NaCl- $\text{Al}_2\text{O}_3$  composite fabricated at 140°C with a holding time of 50 min before and after thermal cycling under a sintering pressure of: (a) 200 MPa, (b) 300 MPa, and (c) 400 MPa.





**Fig. 10** SEM-EDS diagram of NaCl-Al<sub>2</sub>O<sub>3</sub> composite fabricated at 140°C, 400 MPa with a holding time of 50 min: (a) before thermal cycling, (b) after thermal cycling.



**Fig. 11** (a) Relative density variation of NaCl-Al<sub>2</sub>O<sub>3</sub> composite as a function of holding time, (b) Sintered strength variation of NaCl-Al<sub>2</sub>O<sub>3</sub> composite as a function of holding time, and (c) Logarithm of sintered NaCl-Al<sub>2</sub>O<sub>3</sub> composite strength as a function of logarithm of fractional density variation.

water as sintering agent. The composite has good chemical compatibility and stability at high temperature after 30 thermal cycles and holding it at 850°C for 24 h without leakage. The composite demonstrates high mechanical strength, and stable and preserved  $\text{Al}_2\text{O}_3$  structure. The mass loss only occurs at a temperature above 850°C due to volatilization of the chloride salts. In comparison with other fabrication methods, present novel CSP approach demonstrates a new route to fabricate composite material for wide range temperature thermal energy storage and allow material integration to target specific properties for the desired application. The initial understanding of the parameter-structure-property is unveiled, providing fundamental guidance for performance improvement for wide temperature range TES material fabrication.

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